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Temporal Stability of Oxidizers: A Preliminary Survey of Aqueous Solutions

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Aqueous solutions (nominal 0.1N each anion) of various hypochlorite salts (sodium, calcium, lithium) and peroxygen compounds (sodium perborate, sodium persulfate, hydrogen peroxide and magnesium monoperoxyphthalate) have been examined for long-term stability. Lithium hypochlorite solutions in distilled water were stable (i.e., maintained oxidizer capacity) for over 14 days, whereas calcium hypochlorite solutions exhibited oxidizer capacity for only some 5 days. The peroxygen compound solutions displayed stabilities ranging from 2 days (sodium perborate) to approximately 10 days (sodium persulfate and hydrogen peroxide). These solution stability data, monitored by iodometric titrimetry, provide information on how long potential decontamination materials may remain stable in storage as dry, bulk substances. Long stability in solution argues for long stability in bulk form. Stability in storage would be a desirable characteristic, should one or more of the candidates examined prove efficacious for decontamination applications.

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CONTENTS

TEMPORAL STABILITY OF OXIDIZERS:
A PRELIMINARY SURVEY OF AQUEOUS SOLUTIONS

I. INTRODUCTION

Naval operations may be challenged by Chemical Warfare/ Biological Warfare (CW/BW) agents in times of conflict. Any element or aspect of the Navy's operation impacted by such toxic materials has its mission capability severely compromised until such toxics are neutralized. The impacted Naval elements must be decontaminated. Decontamination (decon) of toxic surfaces can be accomplished by passive or active means. As an example, a field commander can choose to allow contamination to weather away by rainfall, sea spray runoff, or solar irradiation, all rather passive decon techniques. However, natural weathering of toxics could be too slow, or even nonexistent, so that use of an active decon would be more effective in returning the compromised element to full military readiness.

Active decon, on the other hand, requires the participation of the occupants of the impacted platform, e.g., ship, etc. Active decon can involve a variety of procedures, ranging from simple washdown of exterior surfaces with seawater through a ship's firefighting system, to use of chemically active countermeasures that chemically attack the toxic material which may be compromising components of the impacted platform. The majority of toxic materials and BW/CW agents will succumb to an oxidation/hydrolysis attack, if appropriate chemical additives are included in the washdown water. Washdown could require large volumes of decon fluid, as in decontaminating a carrier flight deck, or smaller volumes, as for decon of passage ways.

Historically, active decon of CW agents has relied on solutions of hypochlorite in water because of the excellent oxidizing power of hypochlorite ion. Aqueous hypochlorite consists of various proportions of hypochlorite ion (predominant at pH >8) and hypochlorous acid (predominant at pH <6). This fact is significant, as it allows one to tailor a hypochlorite based decon solution to be especially effective against CW agents, or BW materials. Specifically, hypochlorite ion in more basic solutions serves to catalytically hydrolyze, and oxidize, the G-agents, a class of CW compounds (Zirin, et al., 1965; Block and Davis, 1978). Additionally, the use of hypochlorous acid in aqueous solutions at pH <7 (Pellenbarg, 1983) appears effective in countering BW materials such as spores (Hoffman and Spiner, 1962; Fielding, et al., 1967,

1

observations on the use of hypochlorite in BW decon. Overall, decon with aqueous hypochlorite solutions has the potential to be broad spectrum, can be effective against both CW and BW toxics, and is rapid.

The application of aqueous hypochlorite as a decon fluid has emphasized the utility of solid calcium hypochlorite as a source of hypochlorite ion. However, solid calcium hypochlorite disperses slowly in water to give a solution/slurry and the solid tends to decompose in storage. Hypochlorite solutions from a bulk starting material can lead to storage and logistics problems. Thus, it would be advantageous to seek replacement(s) for calcium hypochlorite. Such replacements should exhibit stability in storage if retained as a solid, exhibit ease and completeness of solubility, possess a demonstrated decon effectiveness for CW and BW themselves comparable to that of hypochlorite, and ultimately, lend itself to on-site generation from chemically simple precursors at hand.

Hypochlorite-based decon relying on calcium hypochlorite as a decon fluid component appears as a useful benchmark. There exist, however, serious questions concerning the long-term field deployment of solid hypochlorite: storage suitability, logistics problems, potential corrosivity of the bulk solid, and others. Could decon be augmented or improved by use of materials other than calcium hypochlorite? Needed are data which could guide the selection of a calcium hypochlorite replacement/supplement. The preliminary survey described in this report was designed to examine the stability of potential decon substances in aqueous solution. If, for example, a candidate oxidizer decomposes rapidly in solution (i.e., loses its oxidation power) then one must question the material's potential shelf-life if the candidate oxidizer were to become damp. Such stability information would also be useful when considering the design of packaging for a particular solid oxidizer considered for potential CW/BW decon applications. Naval operations can continue, and can be expected to regain operational status, only if they include dependable, readily available, easily implemented decon protocols for application in a CW/BW threat environment. Thus, the question of decon component (specifically oxidizer/hydrolyzer) stability is fundamental to maintaining an effective operational posture, and is addressed in this preliminary survey.

II. EXPERIMENTAL

Fielding, et al, (1967, 1968) recommended hypochlorite solutions for decon applications as 0.5% (wt. hypochlorite ion/ wt. liquid) in hypochlorite. Thus, 100g of solution should contain 0.5g hypochlorite ion. This solution will be 0.097 Normal [$\approx$0.1 Normal (N)] in hypochlorite ion. All test

solutions (see Table 1) were made up to approximate this nominal <u>oxidizer ion</u> concentration. The resulting solution stability data would be comparable, and could be compared with previous recommendations. The test solutions were made up with house distilled water, and stored in 150 mL Pyrex beakers covered with parafilm during the tests.

Sodium hypochlorite and hydrogen peroxide, available as concentrated solutions, were diluted to the desired final concentration. The other solid oxidizers were weighed out, without drying, as required. None of the test solutions was buffered. This strategy was adopted to more closely resemble what would occur should one of the potential replacement oxidizers be used for preparation of a decon solution in the field. For field use, salts would be used as packaged, and the solutions made up with available water. The tests described later need to be repeated using seawater as the solvent, tests not planned in the current investigation.

The oxidizer content of the various solutions was determined by iodometric titration. (See Pierce and Haenisch, 1948, for a thorough discussion of iodometry). All of the test oxidizers oxidize iodide ion quantitatively. The liberated iodine can be determined by titration with standard thiosulfate reagent. The relevant reactions using hypochlorite ion as the oxidizer example are given in (1) and (2) below:

$$OCl^- + 2H^+ + 2I^- \longrightarrow Cl^- + H_2O + I_2 \qquad (1)$$

$$2 S_2O_3^= + I_2 \longrightarrow S_4O_6^= + 2I^- \qquad (2)$$
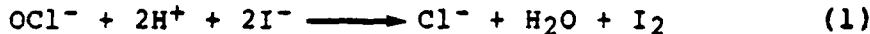
For all test oxidizers, 2.0 mL aliquots of test solution, taken in duplicate as a function of time and measured with a fixed volume disbursing syringe, were combined with ~100 mL distilled water in separate 200 mL beakers. The pH of this solution was adjusted to 4.5 ± 0.5 with 2N H_2SO_4 in distilled water, and 4.0 mL of 20% wt/wt KI in distilled water were added to the reaction beaker with stirring. The resulting solution was titrated to a pale straw color with 0.1 N sodium thiosulfate (Fisher), then 2-3 drops of starch solution were added to give a dark blue solution which was titrated to clear, colorless with thiosulfate. The volume of thiosulfate solution needed to produce a clear, colorless endpoint was recorded. These data are reported in Table 2 as a monitor of oxidizer concentration in a given test solution. Blanks (100 mL distilled water, plus necessary reagents) consumed no thiosulfate, consistently indicating low to zero background oxidants in the house distilled water and reagents as used.

The oxidation half-cell

$$2I^- \rightarrow I_2 + 2e^- \qquad (3)$$

has a potential of -0.53V (CRC Handbook, 1973). Thus, iodide
ion will react in a thermodynamic sense with a broad spectrum
of oxidizer species. Note particularly that in the experiments
described one could consider the iodide ion to be the simulant
for CW/BW toxics. Needed is work which more closely addresses
the interaction of candidate oxidizers with appropriate
simulants or agents, a task not planned within the scope of the
current effort.

This reaction between iodide and hypochlorite was complete
once the reagents were mixed, whereas reactions between the
various peroxocompounds and iodide were noticeably slower. This
fact is significant, and is related to the kinetics of the
reactions between iodide (and presumably any other reducing
agent) and the test peroxygen compounds. Relevant peroxygen
test data are listed in Table 3. A more detailed investigation
of the kinetics of the interactions is also needed. To better
accommodate the reaction kinetics of the iodide-peroxygen
pairs, persulfate and peroxide were allowed to react for ten
minutes with iodide, and the perborate-iodide reaction allowed
to proceed for fifteen minutes before the liberated iodine was
titrated with thiosulfate. These arbitrary reaction times
served as a compromise between seeking complete reaction
between the peroxo ion and iodide, and being able to complete
the required titration in a reasonable amount of time.

III. RESULTS AND DISCUSSION

The titers of thiosulfate solution (0.1 N, nominal),
required to reduce the iodine liberated by the 2.0 mL aliquots
of the various oxidizer solutions tested, are tabulated in
Tables 2 and 3 as a function of the age of the oxidizer
solution. The larger the thiosulfate titer, the more oxidizer
present in the 2 mL aliquot, at any given time in the testing
sequence.

The hypochlorite solutions studied showed (see Figure 1)
both extreme stability with time, as for lithium hypochlorite,
and poor long-term stability, as with calcium hypochlorite.
For fifteen days, the test solution of lithium hypochlorite
exhibited essentially the same thiosulfate titer (~1.7 mL),
whereas the titer for the calcium hypochlorite started at
almost 2.0 mL, and decreased to ~0.40 mL by the end of 15 days.
Sodium hypochlorite was intermediate in long-term stability.
In any case, the solution stability exhibited by the lithium
hypochlorite salt is desirable.

The lithium hypochlorite dissolved easily, and
instantaneously, to give a clear, colorless solution. After
24-36 hours, the solution exhibited a cottony floc. This floc
is postulated to be calcium or magnesium hydroxide or
carbonate, which are both insoluble at the pH (~11.5) of the

4

test solution. The solid lithium hypochlorite that is supplied contains appreciable amounts of magnesium and calcium (i.e., approximately 300 ppm wt/wt each cation, analysis by direct current plasma emission spectrophotometry, Dr. R. Panayappan, NRL). Calcium hypochlorite, on the other hand, dispersed rapidly, and gave a translucent-to-opaque suspension of small particles with an appreciable insoluble residue. The residue, probably calcium carbonate, forms as the bulk calcium hypochlorite interacts with atmospheric carbon dioxide. Note from Table 2 that each of the hypochlorite solutions was initially alkaline. This fact is, in part, due to the tendency of the hypochlorite anion to undergo the following hydrolysis:

$$OCl^- + H_2O \longrightarrow HOCl + OH^- \qquad (4)$$
$$pK = 2.7 \times 10^{-8}$$
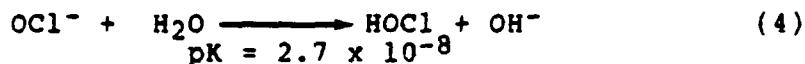
In the case of the calcium hypochlorite solution, which deteriorated the most with time as the hypochlorite ion decomposed, the pH of this solution decreased, reflecting the loss of hypochlorite ion in the solution.

Of the peroxygen compounds studied (see Figure 2), only H-48 exhibited short-term stability in solution. The other peroxygen compounds appeared stable for 2-3 days, and the persulfate was stable in solution for over one week. Data in Table 3 indicate that sodium perborate gave a basic solution, persulfate and peroxide were initially approximately neutral, and H-48 gave a distinctly acidic solution. This behavior is important when considering the potential corrosivity of the solutions tested. Corrosion effects, as a function of peroxygen solute, need to be examined in more detail.

Most peroxygen compounds appear active by release of peroxide ion (HO_2^- as an oxidant, Cotton and Wilkinson, 1972). The perborate, persulfate and H-48 anion also apparently decompose to yield peroxide in solution. This decomposition step is seen in the fact that the peroxygen-iodide oxidation was slow (presumably due to HO_2^- formation first), whereas the hypochlorite-iodide oxidation was very rapid. Of the peroxygen anions studied, only H-48 reacted essentially immediately, and to completion, with the added iodide. However, as the H-48 solution aged, this oxidation rate slowed, so that after four days, the H-48 solution was still oxidizing iodide after 10 minutes, whereas fresh H-48 solution reacted immediately to completion with iodide. Thus, the kinetics of both peroxide ion release, and peroxide ion-simulant/agent interaction appear important in peroxide based decon.

For any decon application, one must recognize that peroxide-bearing or yielding anions may decontaminate more slowly than hypochlorite for two reasons:

5

(1) some peroxygen anions must first decompose to yield the active oxidizer specie; namely, peroxide anion. Indeed, a study of the kinetics of such decomposition is needed. This investigation would yield data useful in elucidating the mechanisms responsible for decon by peroxygen compounds and could point the way toward development of more effective peroxygen decon materials.

(2) peroxide anion has a lower oxidation potential than does hypochlorite:

$$HO_2^- + H_2O + 2e^- \longrightarrow 3\ OH^- \quad E_0 = +\ 0.87 \qquad (5)$$

$$ClO^- + H_2O + 2e^- \longrightarrow Cl^- + 2\ OH^- \quad E_0 = +\ 0.90 \qquad (6)$$
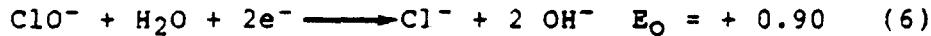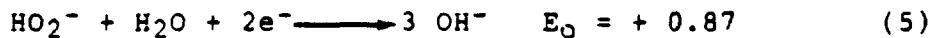
as indicated by the standard cell potential in (5) and (6) (CRC Handbook, 1973). The difference in oxidation potentials, although arguably small, could be significant for decon applications, but may be overshadowed by kinetics.

Of the peroxygen compounds tested, H-48 appears to possess potential merit for decon applications because: the material dissolves rapidly (perborate took on the average 15 minutes to produce 100 mL of 0.1 N solution; W. Bennett, pers. comm., 1984, while H-48 took ~1/2 minute), reacts rapidly with iodide if the H-48 solution in less than 24 hours old, and is a dry granular, free-flowing powder in bulk form. These physical characteristics are important should H-48 compare favorably with hypochlorite as a decon substance. Follow-on work should address this possibility, and directly compare reactions of hypochlorite and H-48 with the same simulants under the same conditions.

IV. CONCLUSIONS

The preliminary survey described in the report examined two major classes of oxidizers, and measured their stability in aqueous solution. Of the hypochlorites studied, the lithium and sodium hypochlorite solutions were markedly more stable than the calcium hypochlorite solution. The stability of lithium hypochlorite in solution argues that lithium hypochlorite may be the hypochlorite salt of choice for potential decon solutions to be prepared from a dry, bulk starting material, since the solid lithium hypochlorite appears very stable in storage.

Decon based on hypochlorite ion should consider the use of lithium hypochlorite if one chooses to make solutions from a bulk, dry starting material. The solution stability exhibited by lithium hypochlorite suggests the potential for a similar stability for solid lithium hypochlorite in storage. Solutions of sodium hypochlorite, however, were almost as stable as those

of lithium hypochlorite, and would be useful for decon as sodium hypochlorite solutions can be prepared in the field electro-chemically from seawater. No bulk starting materials would be required in such an instance. The economics associated with these options should be pursued.

The peroxygen compounds tested appeared less stable in solution than in lithium or sodium hypochlorite. Hydrogen peroxide exhibited marked decomposition between days 4 and 8 (Figure 2) and could serve as a chemical standard when examining other potential peroxygen compounds for efficacy of decon against simulants/agents. Indeed, it appears that peroxygen anions decompose to peroxide anion during the decon process. As hydrogen peroxide is a liquid, its use in the field could prove troublesome. In any case, the current data indicate that H-48, which contains a complex organoperoxide anion, shows promise for decon applications. Reactions with iodide are rapid, and H-48 exhibits positive physical characteristics (dry, non-hygroscopic, free-flowing granular powder). Materials such as perborate may be less useful for decon, as the salt tested required an inordinate amount of time to dissolve. However, the kinetics of peroxide based decon may prove significant. Specifically, persulfate, perborate, and aged solutions of H-48 all exhibited slow reaction with iodide, with the reactions continuing for longer than fifteen minutes. Thus, one could envision a situation with low concentrations of peroxide continuously appearing in a decon solution based on a peroxygen anion. The efficacy of such an approach needs to be investigated in more detail.

V. RECOMMENDATIONS

This initial survey and study of oxidizer solution stability has identified several questions which impact directly on the utility of aqueous oxidizers for decon application. Specifically needed are data which:

(1) Compare the oxidizing ability of candidate oxidizers interacting with species more chemically related to CW, and especially BW, toxins than is iodide ion. Hypochlorite ion and hydrogen peroxide could serve as references for testing purposes in this context.

(2) Examine the solution/decomposition kinetics of peroxygen compounds yielding peroxide anion. Such kinetics studies could yield a new approach to decon where agent on an impacted surface is attacked for a period of time greater than is possible with the more kinetically reactive hypochlorites. Peroxide would be an example of decon candidate which could react more slowly with simulants/toxins to give a longer lived, more gentle decon solution. Peroxide precursors should be addressed in such testing, also.

(3) Investigate the corrosivity associated with solutions of various peroxygen and hypochlorite species. Such solutions should re based on both fresh and seawater.

(4) Examine the stability of the bulk solid oxidizer species in environmentally significant conditions, as functions of temperature and humidity.

(5) Compare the data produced by this study done in fresh water with what could occur if the test compounds were mixed into seawater. Needed are data concerning solution stability, kinetics of candidate/simulant interactions, and so forth, in a seawater matrix.

(6) Address the economics of on-site hypochlorite generation from seawater, as compared with using a bulk starting material for preparation of decon fluids. Such factors as equipment/material storage and use logistics, time for decon solution preparation, application logistics for finished solutions, and others, need to be considered in the overall economics situation.

VI. ACKNOWLEDGMENTS

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Table 1. Oxidizers Tested

Name	Formula Wt	Concentration Calculated Oxidizer Normality	Salt g/ℓ
Sodium Hypochlorite[a] NaOCl	74.5	0.093	6.93
Lithium Hypochlorite[b] LiOCl	58.4	0.065	3.80
Calcium Hypochlorite[a] $Ca(OCl)_2$	143.1	0.142	10.16
Sodium Perborate[c] $NaBO_3 \cdot 4\ H_2O$	153.8	0.098	15.20
Sodium Persulfate[c] $Na_2S_2O_8$	238.0	0.099	23.60
Hydrogen Peroxide[a] H_2O_2	34.0	0.09	3.06
Magnesium Bis[d] (2-Carboxylato Monoperoxybenzoic Acid), Hexahydrate (H-48) Mg $C_{16}H_{24}O_{16}$	496.3	0.11	28.10

[a] Fisher Scientific Company, Fairlawn, NJ
[b] Lithium Corporation of America, Bessemer City, NC
[c] Alfa Products, Danvers, MA
[d] Interox America, Houston, TX

Table 2. Data for Hypochlorite Salts Tested

A. Lithium Hypochlorite (LiOCl)

time (hrs)	pH	titer (mL)
0	11.8	1.70
1	11.8	1.57
5	10.7	1.57
18	11.7	1.70
114	9.5	1.65
264	9.5	1.55
336	9.4	1.95

B. Calcium Hypochlorite [Ca(OCl)$_2$]

time (hrs)	pH	titer (mL)
0	12.5	1.95
5	10.1	1.85
24	9.2	1.60
118	8.4	1.70
270	7.7	0.80
336	7.8	0.80
504	7.1	0.40

C. Sodium Hypochlorite (NaOCl)

time (hrs)	pH	titer (mL)
0	10.4	2.40
1	-	2.40
6	9.9	2.25
24	9.7	2.00
120	9.2	2.30
270	9.4	2.10
340	9.5	2.30
504	9.3	2.00

11

Table 3. Data for Peroxygen Salts Tested

A. Sodium Perborate ($NaBO_3$)

time (hrs)	pH	titer (mL)
0	11.2	1.20
4	11.2	1.25
24	10.8	1.40
48	10.5	1.15
72	10.5	0.85
96	10.3	0.70
168	10.4	0.40
264	10.3	0.20

B. Sodium Peroxydisulfate ($Na_2S_2O_8$)

time (hrs)	pH	titer (mL)
0	6.8	0.50
3	6.9	0.30
24	7.2	0.50
48	6.8	0.50
72	3.6	0.42
95	2.5	0.50
168	2.2	0.55
264	1.5	0.45

C. H-48 (Mg $C_{16}H_{24}O_{16}$)

time (hrs)	pH	titer (mL)
0	3.7	2.20
3.5	3.1	2.10
24	2.8	0.60
49	2.8	0.20
72	2.9	0.18
96	2.8	0.15
168	2.9	0.10
264	2.7	0.37

Table 3.   (continued)

D.   Hydrogen Peroxide (H_2O_2)

time (hrs)	pH	titer (mL)
0	7.7	0.90
3.5	7.7	0.90
24	8.3	1.10
49	8.4	0.90
72	8.5	0.80
96	8.4	0.80
168	8.7	0.40
264	8.9	0.22
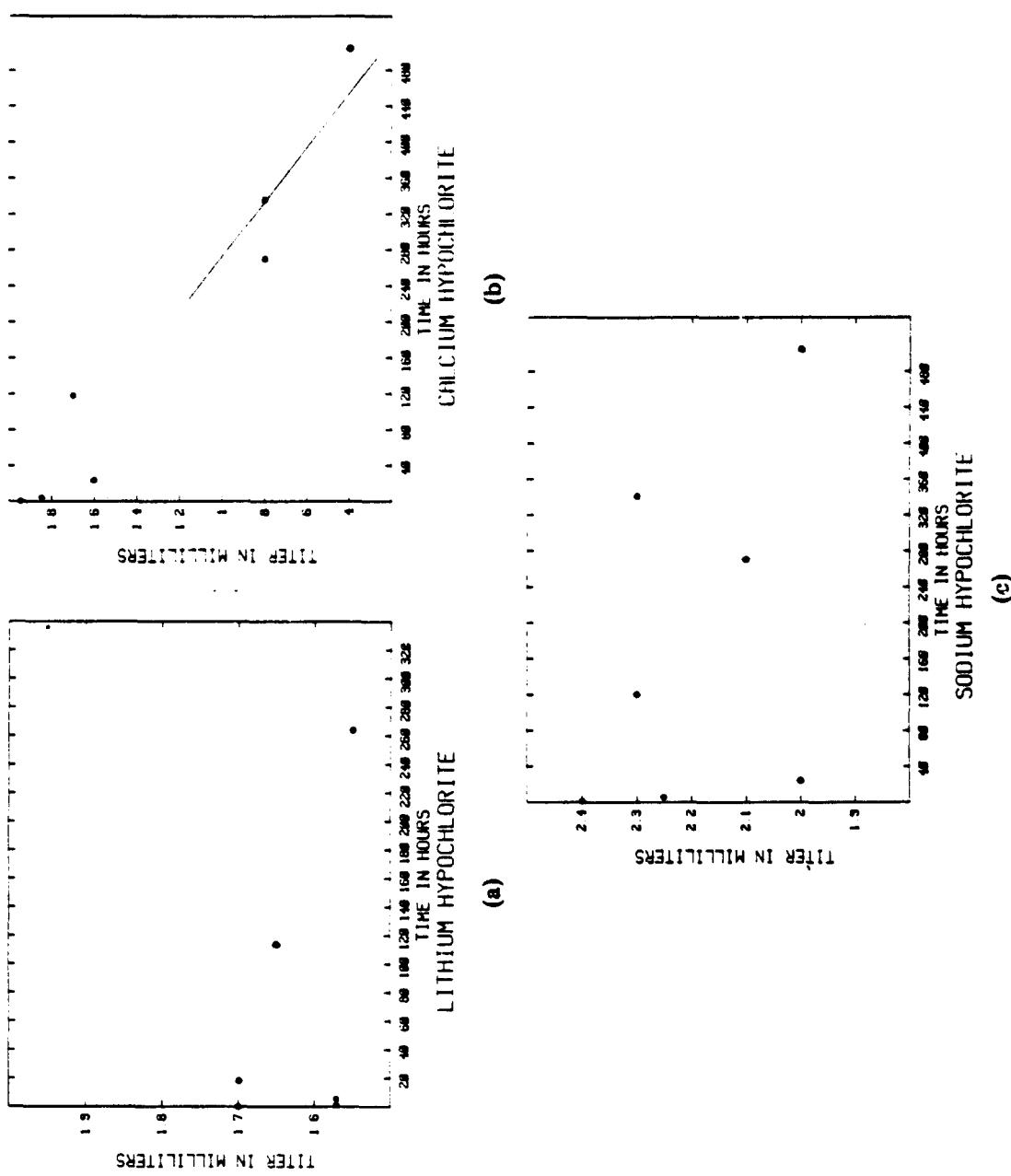
Figure 1. Titers as a function of time for the hypochlorites examined.

14
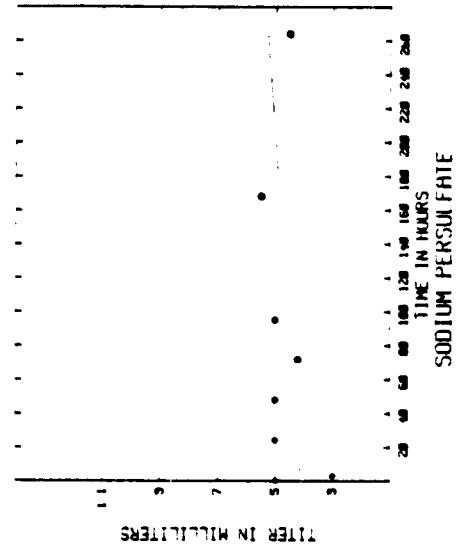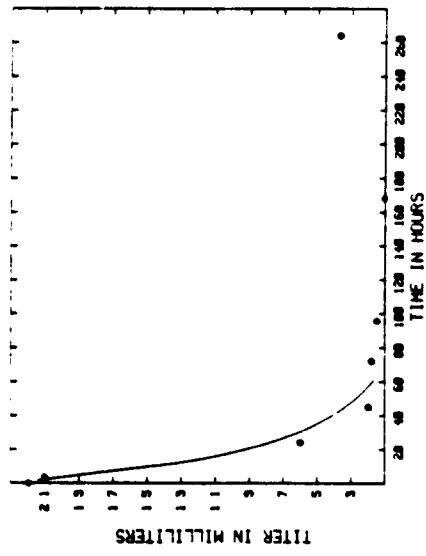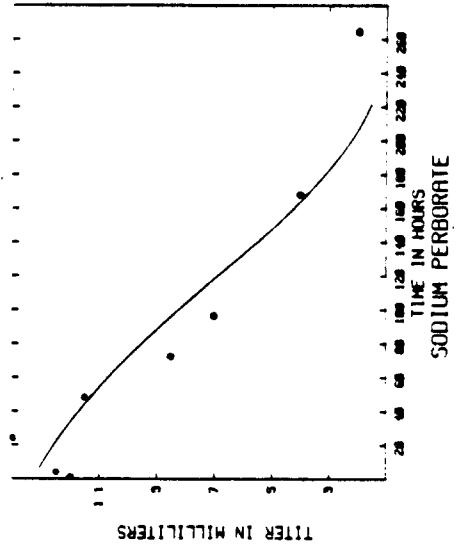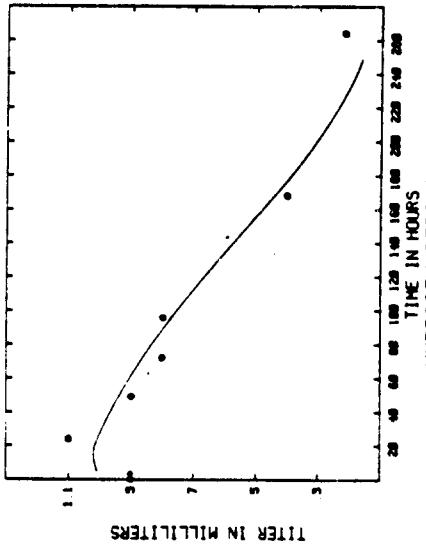
Figure 2. Titers as a function of time for the peroxygen compounds examined.

15